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<p>[54] Title: Aqueous powder coating dispersion</p>		

auxiliaries and other additives such as de-gassing agents, UV absorbers, radical traps and/or antioxidants is mixed together and placed into an emulsifying device, preferably together with water and stabilizers, and in that the resultant emulsion is cooled off and filtered.

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Aqueous Powder Coating Dispersion

The present invention relates to an aqueous powder coating dispersion that is particularly well-suited as a top coat for automobile bodies coated with a water-based paint.

When it comes to the coating of automobile bodies, preference is given to liquid paints nowadays. These entail numerous environmental problems because of the solvents they contain. This is also true when water-based paints are used.

Up until now, completely solvent-free dispersion paints have only been used in areas of application (for instance, as wall paints in building protection) that have relatively simple requirements in terms of the resulting surface properties. In those sectors where the coating has to meet very high visual and mechanical standards, for example, in coil coating or for automotive top coats, it is currently not possible to completely do without organic solvents. Thus, many water-based paints have a residual content of organic solvents within the range from 10% to 20%, as a result of which they should actually be referred to as solvent-reduced coating systems.

For this reason, there have been increased efforts in recent years to employ powder paints for coating jobs. The results, however, are not yet satisfactory, particularly since thicker layers are needed in order to achieve a uniform appearance. Moreover, coating films on a powder basis are not yet optimal in terms of their visual properties such as the flow and gloss or of their mechanical-technical properties such as resistance to chemicals, to weathering and to water drops. Furthermore, the cross-linking reactions suitable for the line conditions of powder coating operations in the automotive sector have some weak points with respect to chemical and scratch resistance.

of powder coating technology in existing coating lines is often not economically feasible and consequently the conversion to solvent-free coating systems is postponed until completely new coating installations are built. Presently, in view of the relatively large particle size of about 25 μm , powder coating technology is hardly able to create coating films that have a layer thickness of less than 50 μm .

The installations for liquid coatings cannot be used for powder coatings. On the other hand, liquid coatings have the above-mentioned drawbacks. In particular, the more environmentally friendly water-based coating systems are only a compromise between the solvent content and coating quality. In many cases, the film properties can only be improved upon at the expense of a higher solvent content. For this reason, efforts are under way to develop powder coatings in the form of aqueous dispersions which can be processed by means of liquid coating technology (U.S. patent no. 3,737,401, U.S. patent no. 3,787,230, German published examined application no. 2,601,618, German preliminary published application no. 2,140,843, German patent no. 2,716,118, U.S. patent no. 4,477,530, U.S. patent no. 4,686,249 BASF F+F AG (1987), U.S. patent no. 4,510,275, U.S. patent no. 4,122,055, U.S. patent no. 4,385,138, WO 96/37,561).

These so-called powder slurries are stable aqueous dispersions of powder coatings. The first powder slurries were prepared by suspending powder coatings in water. The size of the powder coating resin particles ranged from 0.5 μm to 80 μm . It was possible to attain coating formulations with a solids content in the range from 20% to 70%. In contrast to water-based coatings, the addition of an organic solvent as a flow agent is not necessary for powder slurries, since the aqueous phase allows the coating particles to relax well after the coating has been applied. This is possible because, at that point in time, the resin particles are present in a relatively low-viscosity medium and thus display sufficient

powder slurry has been prepared in an improved manner.

powder coating. As far as the particle size of the resin is concerned, however, the systems differ greatly from each other, which is reflected by the application of the powder slurries at a lesser layer thickness (20 μm to 45 μm) of the stoved paint. Therefore, the advantage of powder coatings, namely, the separate timing of the melting of the resin particles to form a smooth film and the subsequent reaction to form a close network, is found in the powder slurry systems as well.

Powder slurries and their preparation are the subject of numerous publications and patent applications. Thus, German preliminary published application no. 2,140,843 describes the preparation of a powder coating by means of spraying or injecting a coating into water, separating and drying the precipitated powder. As an alternative to this preparation method, an attempt was made to convert the powder coating into powder slurries by means of mechanical pre-comminution, followed by fine wet-grinding in water.

Moreover, a process is known from U.S. patent no. 4,268,542 in which a powder coating slurry is used that is suitable for the coating of automobiles. Here, first of all, a commonly employed powder layer is applied onto the body of the automobile, followed by a second layer consisting of clear-lacquer slurry. High stoving temperatures (above 160°C [320°F]) are needed to process this clear-lacquer slurry.

U.S. patent no. 5,379,947 describes a powder slurry system for the coating of automobiles that is based on a hydroxy-functional binder and on an ϵ -caprolactam-blocked isophorone-diisocyanate cross-linking agent. Once the powder slurry has been applied by means of a pneumatic-electrostatic technique, the slurry is first dried for 10 minutes at 49°C [120.2°F] and then stoved for 30 minutes at 177°C [350.6°F].

EXAMPLES OF THE INVENTION

the primers, in addition to which the thickness of the polyurethane is 100 μm .

The production processes described in the above-mentioned patents on powder slurry are based on the micronization of solid resins. These are first pre-mixed in a solids mixer, then homogenized in an extruder, pulverized with a mill and subsequently wet-ground in agitator mills to the final particle size in the range from 3 μm to 20 μm ; this is done in water with the addition of several additives such as wetting agents and dispersants.

This method is not only very complex and thus malfunction-prone, but it also entails restrictions that have to be taken into account already during the synthesis of the binders and the cross-linking agents. For instance, the glass transition temperature of the solid resin must not fall below a certain value so as to ensure sufficient caking, which is one of the prerequisites for the micronization and problem-free storage of the pulverized resin. The upper limit value of the glass transition temperature is determined by the requirement for a very good flow at low stoving temperatures, in other words, high reactivity on the part of the combination consisting of binder and cross-linking agent.

Therefore, the present invention has the objective of providing a process for the preparation of an aqueous powder coating dispersion that can be applied onto automobile bodies using existing liquid coating technology and, in particular, that can be already be stoved at temperatures of less than 150°C [302°F].

This objective is achieved in that a liquid melt containing binders and cross-linking agents and possibly also catalysts, auxiliaries and other additives such as de-gassing agents, UV absorbers, radical traps and/or antioxidants is mixed and placed into an emulsifying device, preferably under the addition of water and stabilizers, after which the resultant emulsion is cooled off and filtered.

polymeric components are fed into the dispersing device in the form of viscous resin melts.

For this purpose, the binders and cross-linking agents have to be melted. The ratio of cross-linking agents to binders ranges from 0.6 to 1 : 1.4, preferably 0.8 : 1 to 1 : 1.2. Preferably, the cross-linking agents and binders are heated in separate containers. In this context, the temperature is selected in such a way that both components are melted and their viscosities allow further processing, especially transport. The higher the temperature of the melt, the lower the viscosity and thus the better the mixing qualities that can be achieved. However, a cross-linking reaction has to largely be ruled out. The cross-linking reaction takes place much more rapidly at higher temperatures. For this reason, there is only a narrow temperature-time window in which the available retention time is sufficiently long for the cooling off while concurrently permitting a good emulsification. In order to utilize this in the most efficient manner possible, the binder melt and the cross-linking agent melt are only combined immediately upstream from the mixing zone.

Prior to conveying the components through the installation, steam can be advantageously employed to heat the unit to the desired process temperature. Afterwards, the molten binder-additive mixture and the cross-linking agent melt are conveyed by pumps through the entire installation, preferably in separate, heated feed lines and then metered into a mixer. The throughput rate of the feed pumps can be used to establish a stoichiometric ratio of binder-additive mixture to cross-linking agent melt. Preferably, the ratio of latent OH groups of the acrylate to the NCO groups of the curing agent is 0.6 to 1 : 1.4, preferably 0.8 : 1 to 1 : 1.2.

The liquid mixture is subsequently immediately emulsified in water. In this process, an emulsifier is added to the organic phase and/or to the water. If an aqueous emulsifier-stabilizer solution is used, then the mixture is heated under pressure to a temperature close

The mixing and emulsifying operations can be carried out in two separate machines (Figure 2) or in a single multi-stage machine (Figure 3). For cross-linking reasons, the second solution entails clear-cut advantages since the retention time at the high temperatures is minimized in this case. Any organic solvent still present can be subsequently separated from the aqueous phase by means of vacuum distillation at low temperatures.

Following the emulsification, a cooling operation is immediately performed. This has to be done in such a way that, on the one hand, the dispersed resin particles do not stick together and, on the other hand, the retention time until the point in time when a cross-linking reaction can no longer occur is as short as possible. This objective can be achieved, for instance, by using a heat exchanger, by spraying cold water to bring about cooling or by injecting the emulsion into cold water.

In order to rule out cross-linking reactions during the mixing, emulsifying and cooling phases, the retention time has to be kept as short as possible from the beginning of the mixing phase until the end of the cooling phase. This retention time amounts to less than 5 seconds, preferably less than 1 second. This is why preference is given to continuous methods. For this purpose, machines are used such as rotor-stator dispersion devices (toothed colloid mills or wet-rotor mills, crown gear dispersion machines, intensive mixers) as well as static mixers (Figure 1).

These machines can be employed in the configurations shown in Figures 2 and 3. In Figure 2, the mixing and comminution step is carried out in two different machines. Rotor-stator aggregates as well as, preferably, static mixers, are used for the mixing, whereby the correct selection of the process parameters ensures sufficient mixing intensity while concurrently entailing the lowest possible input levels of energy and heating, that is to

say, the lowest possible retention time at high temperatures.

de l'élément de la machine à haute température, et de l'élément de la machine à basse température.

improvement of the local performance level is possible through the above-mentioned use of a high-pressure homogenizer. Here, the emulsion is pressed through fine openings at pressures ranging from 100 to 1,500 bar, preferably 100 to 1,000 bar, especially 100 to 200 bar, which brings about a marked reduction of the droplet size and thus a better stability of the emulsion during storage.

The presented micronization variants yield solvent-free dispersions having mean particle sizes ranging from 100 to 10,000 nm, preferably 150 to 6,000 nm, especially preferred 400 to 4,000 nm, highly preferred 600 to 3,500 nm and thus considerably finer particles than the aqueous dispersions (particle size ranging from 3 to 20 μm) that can be achieved by wet-grinding of powder coatings according to the state of the art.

Even though emulsion polymerization according to the state of the art results in fine polymer dispersions, these have very high molecular weights ($> 100,000$ g/mole) as a rule. A high molecular weight, however, leads to a lower diffusion rate of the polymer particles during the stoving phase, as a result of which, in turn, there can be flow defects in the coating film. According to the invention, however, the molecular weights range from 1,000 to 20,000 g/mole, preferably 1,000 to 10,000 g/mole, especially preferred 1,500 to 6,000 g/mole, most highly preferred 1,500 to 4,000 g/mole, so that the mentioned disadvantage of a lower diffusion rate does not occur.

Examples of possible state of the art binders for the powder coating dispersions according to the invention are polyacrylates, polyurethanes, aminoplastic resins.

Likewise, it is also possible to employ binders containing epoxide. Consequently, according to the invention, an aqueous powder coating dispersion can also be used that

- a) at least one binder containing epoxide with a content of 30% to 45%, preferably 30% to 35%, of monomers containing glycidyl, optionally with a content of vinyl-aromatic compounds, preferably styrene,
- b) at least one cross-linking agent, preferably straight-chain, aliphatic dicarboxylic acids and/or carboxy-functional polyesters, and
- c) optionally catalysts, auxiliaries, additives that are typical for powder coatings such as de-gassing agents, flow agents, UV absorbers, radical traps, antioxidants,

and

component B is an aqueous dispersion containing

- a) at least one non-ionic thickening agent and
- b) optionally catalysts, auxiliaries, anti-foaming agents, dispersion auxiliaries, wetting agents, preferably carboxy-functional dispersants, antioxidants, UV absorbers, radical traps, small amounts of solvent, flow agents, biocidal agents and/or water-retention agents.

Dispersions having such a composition are used especially for powder clear lacquers.

Examples of suitable epoxy-functional binders for the solid powder coating that is used for the preparation of the dispersion are polyacrylate resins containing epoxide groups that can be produced by means of the copolymerization of at least one ethylenically unsaturated monomer that contains at least one epoxide group in the molecule with at least another ethylenically unsaturated monomer that does not contain an epoxide group in the molecule, whereby at least one of the monomers is an ester of acrylic acid or of methacrylic acid. Such polyacrylate resins containing epoxide groups are known, for instance, from EP-A 299,420, DE-B 2,214,650, DE-B 2,749,576, US-A 4,091,048 and

Examples of ethylenically unsaturated monomers that do not contain an epoxide group in the molecule are alkyl esters of acrylic acid and methacrylic acid having 1 to 20 carbon atoms in the alkyl radical, especially methyl acrylate, methyl methacrylate, ethyl acrylate, ethyl methacrylate, butyl acrylate, butyl methacrylate, 2-ethylhexyl acrylate and 2-ethylhexyl methacrylate. Other examples of ethylenically unsaturated monomers that do not contain an epoxide group in the molecule are acid amides such as, for instance, acrylic acid amide and methacrylic acid amide, vinyl-aromatic compounds such as styrene, methyl styrene and vinyl toluene, nitriles such as acrylonitrile and methacrylonitrile, vinyl halides and vinylidene halides such as vinyl chloride and vinylidene fluoride, vinyl esters such as, for instance, vinyl acetate and monomers containing hydroxyl groups such as, for instance, hydroxyethyl acrylate and hydroxyethyl methacrylate.

The polyacrylate resin containing epoxide groups normally has an epoxide equivalent weight ranging from 400 to 2,500, preferably 420 to 700, a number average molecular weight (determined by means of gel permeation chromatography using a polystyrene standard) of 2,000 to 20,000, preferably 3,000 to 10,000, and a glass transition temperature (T_g) ranging from 30°C to 80°C [86°F from 176°F], preferably from 40°C to 70°C [104°F to 158°F], especially preferred 40°C to 60°C [104°F to 140°F] (measured by means of Differential Scanning Calorimetry DSC). Very special preference is given to a temperature of about 50°C [122°F]. Mixtures of two or more acrylate resins can also be used.

The polyacrylate resin containing epoxide groups can be prepared according to well-known methods by means of polymerization.

Examples of possible cross-linking agents are all of the state of the art compounds known to those skilled in the art, including, but not limited to, the following: particularly,

the ready-made powder clear lacquers, other cross-linking agents containing carboxyl groups can optionally be employed as well. Examples of these are saturated branched or unsaturated straight-chain dicarboxylic acids and polycarboxylic acids as well as polymers with carboxyl groups.

Furthermore, powder coatings are also suitable that contain an epoxy-functional cross-linking agent and an acid-functional binder.

Examples of suitable acid-functional binders are acidic polyacrylate resins which can be prepared by means of copolymerization of at least one ethylenically unsaturated monomer that contains at least one acid group in the molecule with at least another ethylenically unsaturated monomer that does not contain an acid group in the molecule.

The binder containing epoxide groups or the cross-linking agent containing epoxide groups and the carboxylic agent or the binder are normally used in such an amount that, per equivalent epoxide group, there are 0.5 to 1.5, preferably 0.75 to 1.25 equivalent carboxyl groups. The quantity of carboxyl groups present can be determined by means of titration using an alcoholic KOH solution.

According to the invention, the binder can contain vinyl-aromatic compounds, especially styrene. However, in order to limit the risk of crack formation, their content does not lie above 35% by weight. Preference is given to 10% to 25% by weight.

The solid powder coatings optionally contain one or more suitable catalysts for the curing. Suitable catalysts are phosphonium salts of organic or inorganic acids, quaternary ammonium compounds, amines, imidazole and imidazole derivatives. The catalysts are

Examples of suitable phosphonium catalysts are ethyl triphenyl phosphonium iodide, ethyl triphenyl phosphonium chloride, ethyl triphenyl phosphonium thiocyanate, ethyl triphenyl phosphonium acetate acetic acid complex, tetrabutyl phosphonium iodide, tetrabutyl phosphonium bromide and tetrabutyl phosphonium acetate acetic acid complex. These as well as other suitable phosphonium catalysts are described, for example, in U.S. patent no. 3,477,990 and U.S. patent no. 3,341,580.

Examples of suitable imidazole catalysts are 2-styrylimidazole, 1-benzyl-2-methylimidazole, 2-methylimidazole and 2-butyylimidazole. These as well as other imidazole catalysts are described, for instance, in Belgian patent no. 756,693.

Moreover, the solid powder coatings can optionally contain auxiliaries and additives as well. Examples of these are flow agents, antioxidants, UV absorbers, radical traps, pouring auxiliaries and de-gassing agents such as, for instance, benzoin.

Suitable flow agents are those on the basis of polyacrylates, polysiloxanes or fluorine compounds.

Antioxidants that can be employed here are reducing agents such as hydrazides and phosphorus compounds as well as radical traps, for instance, 2,6-di-*tert*-butyl phenol.

UV absorbers that can be used are preferably triazines and benzotriphenol.

Suitable radical traps are 2,2,6,6-tetramethyl piperidine derivatives.

As another component, the aqueous component B of the powder coating dispersion contains a suitable amount of a surface-active agent, for example, a non-ionic surface-active agent.

As a further component, the aqueous component B of the powder coating dispersion contains a suitable amount of a wetting agent, for example, a non-ionic wetting agent.

- aa) a hydrophilic skeleton that ensures adequate water solubility, and
- ab) hydrophobic groups that are capable of associative interaction in an aqueous medium.

Examples of hydrophobic groups that can be used are long-chain alkyl radicals such as, for instance, dodecyl, hexadecyl or octadecyl radicals, or alkaryl radicals such as, for example, octyl-phenyl or nonyl-phenyl radicals.

As hydrophilic skeletons, preference is given to the use of polyacrylates, cellulose ether or, especially preferred, polyurethanes which contain the hydrophobic groups as polymer building blocks.

Especially preferred as hydrophilic skeletons are polyurethanes that contain polyether chains as building blocks, preferably consisting of polyethylene oxide.

In the synthesis of such polyether polyurethanes, the diisocyanates and/or polyisocyanates, preferably aliphatic diisocyanates, especially preferred optionally alkyl-substituted 1,6-hexamethylene diisocyanate, serve to link the polyether building blocks having hydroxyl-group terminals to each other as well as to link the polyether building blocks to the hydrophobic end-group building blocks which can be, for example, monofunctional alcohols and/or amines having the above-mentioned long-chain alkyl radicals or aralkyl radicals.

Furthermore, component B can contain catalysts, flow agents, antioxidants, UV absorbers, radical traps and cross-linking agents. Essentially, the substances already listed for component A are a possibility here, too.

Examples of anti-foaming agents are preferably modified polysiloxanes.

Dispersion auxiliaries are, for instance, preferably ammonium or metal salts of polycarboxylates.

Examples of neutralizing agents that can be used here are amines, ammonia and metal hydroxides. Other preferred binders according to the invention are hydroxy-functionalized compounds, especially acrylates containing hydroxyl groups. Especially preferred are hydroxy-functionalized polymethacrylates.

Other preferred binders according to the invention are hydroxy-functionalized compounds, especially acrylates containing hydroxyl groups. Especially preferred are hydroxy-functionalized polymethacrylates. Examples of such polyhydroxy-functional polyacrylates (polyacrylate polyols) are those which preferably contain hydroxyalkyl esters of acrylic acid, methacrylic acid or another α -ethylenically unsaturated, β -ethylenically unsaturated carboxylic acid as the comonomer units. These esters can be derived from an alkylene glycol that is esterified with an acid, or they can be obtained by reacting the acid with an alkylene oxide.

As the hydroxyalkyl esters, preference is given to the use of hydroxyalkyl esters of (meth)acrylic acid in which the hydroxylalkyl group contains up to 4 carbon atoms, or mixtures consisting of these hydroxyalkyl esters.

Examples of these are 2-hydroxyethyl-(meth)acrylate, 2- or 3-hydroxypropyl-(meth)acrylate or 4-hydroxybutyl-(meth)acrylate.

Examples of cross-linking agents in this case are preferably blocked isocyanates. Thanks to the systematic combination of different polyisocyanate pre-polymers containing differing blocking agents, the reactivity of the cross-linking agent vis-à-vis polymeric bind-

The above-mentioned dispersion auxiliaries or emulsifiers can be employed for stabilization purposes. In addition, it is also possible to use other substances.

In order to ensure that the application and film properties of the powder slurry do not change even after prolonged storage of the material, the binders as well as the cross-linking agents must not be able to react at room temperature with each other or with other additives or with the water that serves as the solvent.

The physical stability is reflected in the settling and agglomeration behavior of the resin particles in the aqueous solution. The fine dispersions consisting of coating powder particles or the micronized reagents present in the powder slurry have a mean particle size ranging from 100 to 10,000 nm, preferably 150 to 6,000 nm, especially preferred 400 to 4,000 nm, highly preferred 800 to 3,500 nm and molecular weights ranging from 1,000 to 20,000 g/mole, preferably 1,000 to 10,000 g/mole, especially preferred 1,500 to 6,000 g/mole, highly preferred 1,500 to 4,000 g/mole, whereby the fine dispersions are not stable without the appropriate surface-active substances and consequently would lose their processing properties and the coating behavior as a result of particle interactions.

In any case, the stabilizers have to have the following properties:

1. They should be very efficient, that is to say, small doses should help to stabilize large quantities of dispersion material.
2. They hydrophilia should be such that they can be "switched", that is to say, it should be possible to convert the hydrophilic groups in the solids into hydrophobic structural components by means of chemical reactions.

3. The chemical structure should be compatible with the coating resin in order to prevent deterioration of the property profile as a result of de-mixing or incomplete build-up.

According to the invention, possible emulsifiers are those on the basis of polyurethane and prepared by means of the co-addition of hydrophilic and hydrophobic polyalcohols and polyesters with diisocyanates and amine lengthening, whereby the substance composition and thus the amphiphilia, the molecular weight and the number of functional group can all be varied. In this context, the amphiphilia is adjusted to the special coating resin-water interface in such a way that an effective and long-term stable dispersion can be achieved with a minimal amount of stabilizer. The remaining functional groups, in turn, are selected in such a way that they are chemically incorporated into the coating film during the curing and thus "de-hydrophilize" the emulsifier.

Alternatively, transferring polymerization in a lyotropic phase can be employed to produce short-chain amphiphilic polyacrylates from acrylic acid, ethyl hexyl acrylate, hydroxyethyl acrylate and an anionic comonomer. Aside from the urethane chemistry, these systems also allow a "carboxy-epoxy" two-component structure. The powder coating dispersion according to the invention can be used as a pigmented coating in the form of a clear lacquer as a top coating of primers, preferably in the automotive industry.

The powder coating dispersions according to the invention can be applied by means of the methods known from liquid coating technology. In particular, they can be applied by means of spraying techniques. It is likewise possible to employ electrostatically aided high rotation or pneumatic application.

Subsequently, at a slightly elevated temperature (above the glass transition temperature)

amounts to 40°C to 70°C [104°F to 158°F], preferably 50°C to 65°C [122°F to 149°F]. The flashing-off is carried out for 2 to 10 minutes, preferably 4 to 8 minutes at room temperature. Flashing-off is done again for the same period of time at an elevated temperature.

The stoving operation can be already conducted at temperatures of 130°C [266°F]. It is feasible to perform the stoving operations at temperatures ranging from 130°C to 180°C [266°F to 356°F], preferably from 135°C to 155°C [275°F to 311°F].

Below, the invention will be described in greater detail with reference to the examples.

- I. Aqueous powder coating dispersions on the basis of OH-acrylate/blocked polyisocyanate prepared by dispersing a powder coating in water and by subsequently setting the particle size by means of wet-grinding in a sand mill.

- 1) Preparation of the acrylate resin

40 parts of xylene are prepared and heated up to 130°C [266°F]. Then, at a temperature of 130°C [266°F] over the course of 4 hours, the following are metered into the recipient via two separate inlets: initiator 4.5 parts of TBPEH (*tert*-butyl perethyl hexanoate) mixed with 4.86 parts of xylene, and monomer 15 parts of styrene, 20 parts of *n*-butyl methacrylate, 30 parts of cyclohexyl methacrylate, 35 parts of 2-hydroxyethyl methacrylate. Subsequently, the mixture is heated up to 170°C [338°F] and the solvent is extracted in a vacuum < 100 mbar.

- 2) Preparation of the powder clear lacquer

56.07 parts of acrylate resin (OH content of 4.58%), 20.93 parts of Vestanat B

10.00 parts of xylene, 10.00 parts of dibutyltin dilaurate, 10.00 parts of dibutyltin dilaurate

(catalyst) are blended together thoroughly in a Henschel fluid mixer, extruded on a Buss PLK 46 extruder, ground up in a Hosohawa ACM2 mill and sifted through a 125- μ m sieve.

3) Preparation of the dispersion

1.6 parts of Orotan 731 K (dispersion auxiliary), 0.2 parts of Troykyd 777 (anti-foaming agent), 0.4 parts of Surfinol TMN 6 (cross-linking agent), 1.8 parts of Acrysol RM8 (Rohm & Haas) (non-ionic associative thickening agent on a polyurethane basis) as well as 35 parts of the powder clear lacquer prepared according to 2) are dispersed in 61 parts of demineralized water. The material is ground up in a sand mill for 2 hours. The subsequently measured average particle size is 3.6 μ m. The material is filtered through a 50- μ m filter, after which 0.5% Byk 345 (flow agent) is added.

4) Application of the dispersion

A cup gun is used to apply the slurry onto steel plates coated with water-based coating. The plate is flashed-off for 5 minutes at room temperature and for another 5 minutes at 60°C [140°F]. Subsequently, the plate is stoved for 30 minutes at a temperature of 150°C [302°F]. At a layer thickness of 40 μ m, a high-gloss clear lacquer film is produced that has MEK resistance (> 100 up-and-down strokes). The clear lacquer film displays good resistance to condensation water.

II. Powder slurries prepared by mixing melts and their subsequent dispersion in water

1) The powder slurry is preferably prepared as follows:

The powder slurry is prepared by mixing the following components in the following order:

1. A clear lacquer prepared according to the method described in Example 1, 2 or 3, 4 or 5, 6 or 7, 8 or 9, 10 or 11, 12 or 13, 14 or 15, 16 or 17, 18 or 19, 20 or 21, 22 or 23, 24 or 25, 26 or 27, 28 or 29, 30 or 31, 32 or 33, 34 or 35, 36 or 37, 38 or 39, 40 or 41, 42 or 43, 44 or 45, 46 or 47, 48 or 49, 50 or 51, 52 or 53, 54 or 55, 56 or 57, 58 or 59, 60 or 61, 62 or 63, 64 or 65, 66 or 67, 68 or 69, 70 or 71, 72 or 73, 74 or 75, 76 or 77, 78 or 79, 80 or 81, 82 or 83, 84 or 85, 86 or 87, 88 or 89, 90 or 91, 92 or 93, 94 or 95, 96 or 97, 98 or 99, 100 or 101, 102 or 103, 104 or 105, 106 or 107, 108 or 109, 110 or 111, 112 or 113, 114 or 115, 116 or 117, 118 or 119, 120 or 121, 122 or 123, 124 or 125, 126 or 127, 128 or 129, 130 or 131, 132 or 133, 134 or 135, 136 or 137, 138 or 139, 140 or 141, 142 or 143, 144 or 145, 146 or 147, 148 or 149, 150 or 151, 152 or 153, 154 or 155, 156 or 157, 158 or 159, 160 or 161, 162 or 163, 164 or 165, 166 or 167, 168 or 169, 170 or 171, 172 or 173, 174 or 175, 176 or 177, 178 or 179, 180 or 181, 182 or 183, 184 or 185, 186 or 187, 188 or 189, 190 or 191, 192 or 193, 194 or 195, 196 or 197, 198 or 199, 200 or 201, 202 or 203, 204 or 205, 206 or 207, 208 or 209, 210 or 211, 212 or 213, 214 or 215, 216 or 217, 218 or 219, 220 or 221, 222 or 223, 224 or 225, 226 or 227, 228 or 229, 230 or 231, 232 or 233, 234 or 235, 236 or 237, 238 or 239, 240 or 241, 242 or 243, 244 or 245, 246 or 247, 248 or 249, 250 or 251, 252 or 253, 254 or 255, 256 or 257, 258 or 259, 260 or 261, 262 or 263, 264 or 265, 266 or 267, 268 or 269, 270 or 271, 272 or 273, 274 or 275, 276 or 277, 278 or 279, 280 or 281, 282 or 283, 284 or 285, 286 or 287, 288 or 289, 290 or 291, 292 or 293, 294 or 295, 296 or 297, 298 or 299, 300 or 301, 302 or 303, 304 or 305, 306 or 307, 308 or 309, 310 or 311, 312 or 313, 314 or 315, 316 or 317, 318 or 319, 320 or 321, 322 or 323, 324 or 325, 326 or 327, 328 or 329, 330 or 331, 332 or 333, 334 or 335, 336 or 337, 338 or 339, 340 or 341, 342 or 343, 344 or 345, 346 or 347, 348 or 349, 350 or 351, 352 or 353, 354 or 355, 356 or 357, 358 or 359, 360 or 361, 362 or 363, 364 or 365, 366 or 367, 368 or 369, 370 or 371, 372 or 373, 374 or 375, 376 or 377, 378 or 379, 380 or 381, 382 or 383, 384 or 385, 386 or 387, 388 or 389, 390 or 391, 392 or 393, 394 or 395, 396 or 397, 398 or 399, 400 or 401, 402 or 403, 404 or 405, 406 or 407, 408 or 409, 410 or 411, 412 or 413, 414 or 415, 416 or 417, 418 or 419, 420 or 421, 422 or 423, 424 or 425, 426 or 427, 428 or 429, 430 or 431, 432 or 433, 434 or 435, 436 or 437, 438 or 439, 440 or 441, 442 or 443, 444 or 445, 446 or 447, 448 or 449, 450 or 451, 452 or 453, 454 or 455, 456 or 457, 458 or 459, 460 or 461, 462 or 463, 464 or 465, 466 or 467, 468 or 469, 470 or 471, 472 or 473, 474 or 475, 476 or 477, 478 or 479, 480 or 481, 482 or 483, 484 or 485, 486 or 487, 488 or 489, 490 or 491, 492 or 493, 494 or 495, 496 or 497, 498 or 499, 500 or 501, 502 or 503, 504 or 505, 506 or 507, 508 or 509, 510 or 511, 512 or 513, 514 or 515, 516 or 517, 518 or 519, 520 or 521, 522 or 523, 524 or 525, 526 or 527, 528 or 529, 530 or 531, 532 or 533, 534 or 535, 536 or 537, 538 or 539, 540 or 541, 542 or 543, 544 or 545, 546 or 547, 548 or 549, 550 or 551, 552 or 553, 554 or 555, 556 or 557, 558 or 559, 560 or 561, 562 or 563, 564 or 565, 566 or 567, 568 or 569, 570 or 571, 572 or 573, 574 or 575, 576 or 577, 578 or 579, 580 or 581, 582 or 583, 584 or 585, 586 or 587, 588 or 589, 590 or 591, 592 or 593, 594 or 595, 596 or 597, 598 or 599, 600 or 601, 602 or 603, 604 or 605, 606 or 607, 608 or 609, 610 or 611, 612 or 613, 614 or 615, 616 or 617, 618 or 619, 620 or 621, 622 or 623, 624 or 625, 626 or 627, 628 or 629, 630 or 631, 632 or 633, 634 or 635, 636 or 637, 638 or 639, 640 or 641, 642 or 643, 644 or 645, 646 or 647, 648 or 649, 650 or 651, 652 or 653, 654 or 655, 656 or 657, 658 or 659, 660 or 661, 662 or 663, 664 or 665, 666 or 667, 668 or 669, 670 or 671, 672 or 673, 674 or 675, 676 or 677, 678 or 679, 680 or 681, 682 or 683, 684 or 685, 686 or 687, 688 or 689, 690 or 691, 692 or 693, 694 or 695, 696 or 697, 698 or 699, 700 or 701, 702 or 703, 704 or 705, 706 or 707, 708 or 709, 710 or 711, 712 or 713, 714 or 715, 716 or 717, 718 or 719, 720 or 721, 722 or 723, 724 or 725, 726 or 727, 728 or 729, 730 or 731, 732 or 733, 734 or 735, 736 or 737, 738 or 739, 740 or 741, 742 or 743, 744 or 745, 746 or 747, 748 or 749, 750 or 751, 752 or 753, 754 or 755, 756 or 757, 758 or 759, 760 or 761, 762 or 763, 764 or 765, 766 or 767, 768 or 769, 770 or 771, 772 or 773, 774 or 775, 776 or 777, 778 or 779, 780 or 781, 782 or 783, 784 or 785, 786 or 787, 788 or 789, 790 or 791, 792 or 793, 794 or 795, 796 or 797, 798 or 799, 800 or 801, 802 or 803, 804 or 805, 806 or 807, 808 or 809, 810 or 811, 812 or 813, 814 or 815, 816 or 817, 818 or 819, 820 or 821, 822 or 823, 824 or 825, 826 or 827, 828 or 829, 830 or 831, 832 or 833, 834 or 835, 836 or 837, 838 or 839, 840 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Tinuvin 144 (HALS), 0.4 parts of Additol XL 490 (flow agent), 0.4 parts of benzoin (de-gassing agent) and 0.5 parts of dibutyl tin oxide (catalyst) are all heated up to a process temperature of 139°C [282.2°F]. This process temperature is selected in such a way that both components are melted and their viscosities allow further processing, particularly transport.

Prior to transporting the components through the installation, steam is employed to heat the installation up to the process temperature. Then, the molten binder-additive mixture and the cross-linking agent melt are conveyed by pumps through the entire installation in separate, heated lines and then metered into a mixer. The pumps function volumetrically. The throughput rate of the pumps is used to establish a stoichiometric ratio of binder-additive mixture to cross-linking agent melt (OH groups of the acrylate to latent NCO groups of the curing agent = 1:1). The melts are mixed molecularly and disperse within a very short time (< 5 s) in a mixing chamber (static mixer, manufactured by the Sulzer GmbH company). The resulting homogeneous melt, which is still at a temperature above the melting point of the components, is conveyed to a pre-emulsifying zone, where an aqueous solution consisting of 61 parts of demineralized water, 1.6 parts of Orotan 731 K (dispersion auxiliary), 0.2 parts of Troykyd 777 (anti-foaming agent) and 0.4 parts of Surfinol TMN 6 (cross-linking agent) is metered by a pump that functions volumetrically. Prior to being metered in, the aqueous solution was heated up to the process temperature of 139°C [282.2°F] in a pressure-proof container 3. By applying energy by means of a rotor-stator system or a static mixer, a pre-emulsion is then created.

In this context, the resin cross-linking agent melt forms drops (dispersed phase) in the aqueous solution (continuous phase). The drops of the pre-emulsion are reduced to a particle size between 1,000 and 3,000 nm in another zone by means

of a rotor-stator system or a static mixer. The resulting pre-emulsion is then conveyed

Following the dispersion, the dispersions are cooled off by the injection of cold water. The amounts of demineralized water for the pre-emulsification and for cooling off the dispersions are dimensioned in such a way that the resultant dispersion has a solids content of about 35%. Subsequently, 1.8 parts of Acrysol RM8 (thickening agent, relative to a solids content of 35%) are added to the dispersion and filtered through a 50- μ m filter.

2) Application of the dispersion

A cup gun is used to apply the slurry onto steel plates coated with a water-based coating. The plate is flashed-off for 5 minutes at room temperature and for 5 minutes at 60°C [140°F]. Subsequently, the plate is stoved for 30 minutes at a temperature of 150°C [302°F]. At a layer thickness of 40 μ m, a high-gloss clear lacquer film is produced that has MEK resistance (> 100 up-and-down strokes). The clear lacquer film displays good resistance to condensation water.

Patent Claims

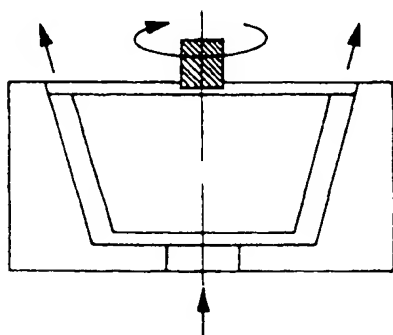
1. Process for the preparation of an aqueous powder coating dispersion, **characterized in that** a liquid melt containing binders and cross-linking agents and possibly also catalysts, auxiliaries and other additives such as de-gassing agents, flow agents, UV absorbers, radical traps and/or antioxidants is placed into an emulsifying device preferably under the addition of water and stabilizers, after which the emulsion thus obtained is cooled off and filtered.
2. Process according to Claim 1, **characterized in that** binders and agents on the one hand, and cross-linking agents on the other hand, are melted down separately, subsequently mixed with each other, after which the dispersion is carried out.
3. Process according to Claim 1 or 2, **characterized in that** the ratio of binder to cross-linking agent in the melt ranges from 0.6 to 1 : 1.4, preferably 0.8 : 1 to 1 : 1.2.
4. Process according to one of Claims 1 through 3, **characterized in that** the melt is mixed for less than 5 seconds, preferably less than 1 second.
5. Process according to one of Claims 1 through 4, **characterized in that** the mixture is pressed through fine openings at pressures ranging from 100 to 1,500 bar, preferably 500 to 1,000 bar.

operation.

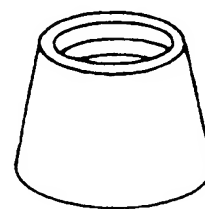
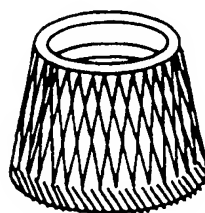
7. Process according to one of Claims 1 through 6, **characterized in that** the cooling is effectuated by means of heat exchangers, spraying the emulsion with water or evaporating the emulsion in water.
8. Process according to one of Claims 4 through 6, **characterized in that** rotor-stator dispersing devices, preferably static mixers as well as toothed colloid mills or wet-rotor mills, crown gear dispersion machines, intensive mixers or high-pressure homogenizers are used.
9. Process according to one of Claims 1 through 8, **characterized in that** polyacrylate resin, polyurethane resin and aminoplastic resins as well as epoxy acrylates, preferably hydroxy-functionalized acrylates, are used as the binders.
10. Aqueous powder coating dispersion, **characterized in that** it can be obtained by means of a process according to one of Claims 1 through 8.
11. Aqueous powder coating dispersion according to Claim 10, **characterized in that** the mean particle size of the powder coating particles lies within the range from 100 to 10,000 nm, preferably 150 to 6,000 nm, and the molecular weight lies between 1,000 and 20,000 g/mole, preferably 1,500 and 4,000 g/mole.
12. Use of the aqueous powder coating dispersion according to either Claim 10 or 12 for

Dispersing machines

Colloid mills (wet rotor mills)

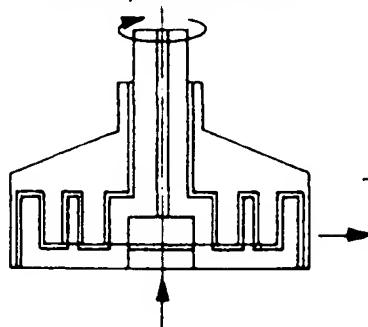


toothed
surfaces of the rotor and stator

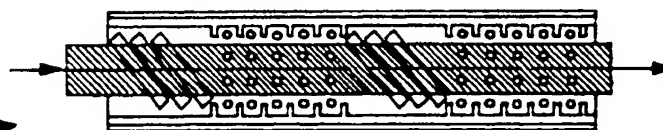


Crown gear dispersion machines / intensive mixers

radial product flow



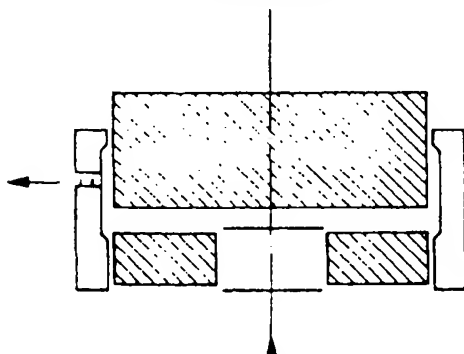
axial product flow



with transport elements

High-pressure homogenizers

"standard"



counterflow principle

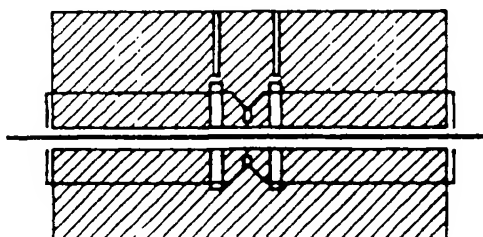


FIGURE 1

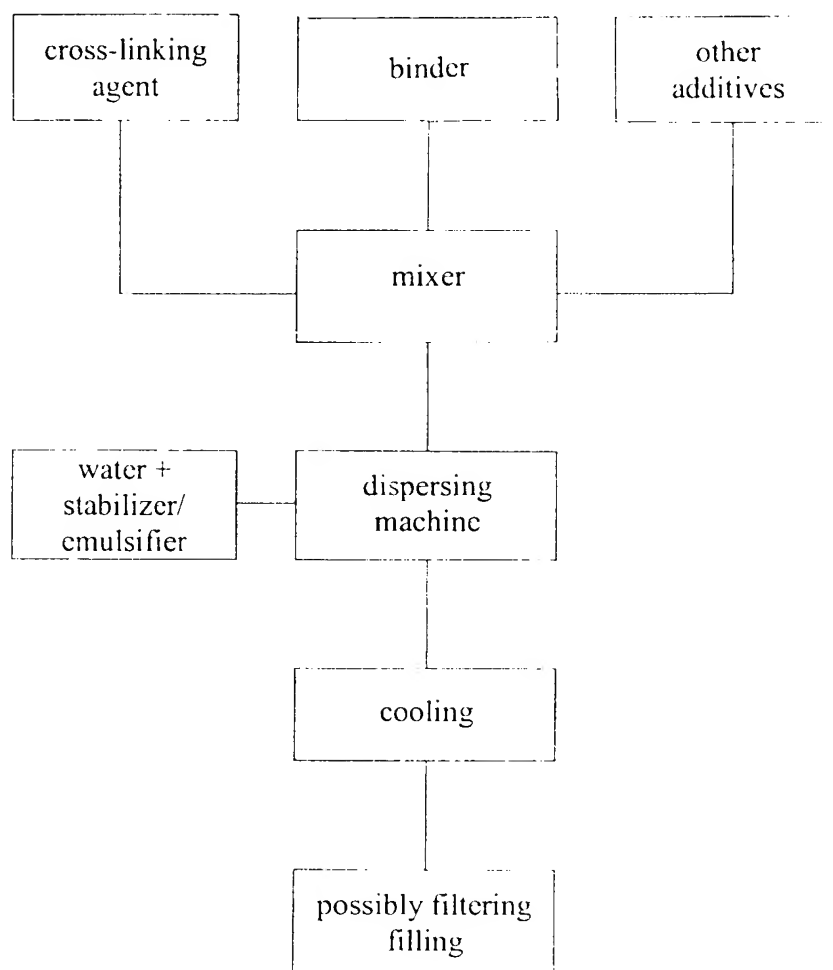


Figure 2: Process variants with separate mixing and emulsifying steps.

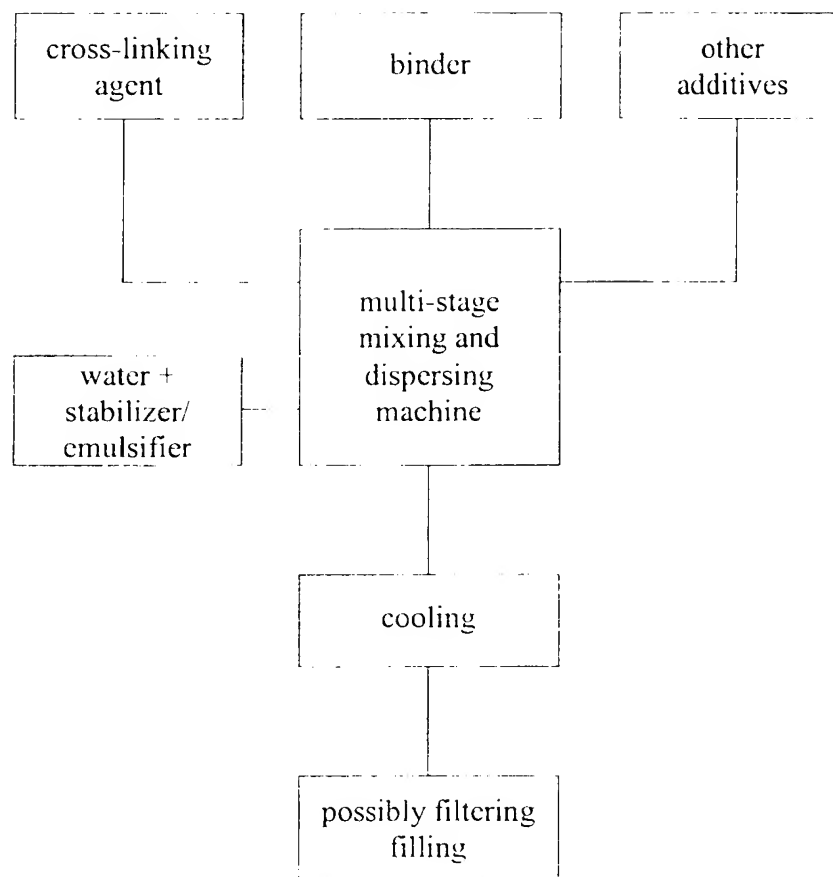


Figure 3: Process variants with combined mixing and emulsifying steps.

Translation: Language Services Unit

Translated from EP 0 800 000 A1